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(Translation)

[DOCUMENT NAME] Specification

[TITLE OF THE INVENTION] Polymer electrolyte fuel cell and method for restoring performance thereof

[CLAIMS]

[Claim 1] A polymer electrolyte fuel cell characterized by comprising:

(a cell body composed of laminated unit cells, each comprising a polymer electrolyte membrane, a fuel electrode and an air electrode sandwiching said polymer electrolyte membrane therebetween and a pair of separator plates having gas flow paths for supplying and discharging an oxidant gas to and from said air electrode and supplying and discharging a fuel gas to and from said fuel electrode);

means for supplying and discharging said oxidant gas and said fuel gas to and from said cell body; and

means for controlling output of a current generated in said cell body; and

means for restoring performance of said cell.

[Claim 2] The polymer electrolyte fuel cell in accordance with claim 1,

characterized in that said separator plates contain a metal material.

[Claim 3] The polymer electrolyte fuel cell in accordance with claim 1 or 2,

characterized in that a contaminant ion accumulated in said cell body is discharged to the outside of said cell body with said means for restoring performance of said cell.

[Claim 4] A method for restoring the polymer electrolyte fuel cell in accordance with claim 1, 2 or 3, characterized by comprising

operating said polymer electrolyte fuel cell in a loaded current mode different from that of a normal operation for a predetermined time, thereby restoring performance of said cell.

[Claim 5] The method for restoring performance of a polymer electrolyte fuel cell in accordance with claim 4,

characterized in that said loaded current mode different from that of the normal operation is either an operation mode at a current of not less than 1.5 times as high as that in the normal operation or an operation mode at a current giving an output voltage per unit cell of not more than 0.2 V.

[Claim 6] A method for restoring performance of the polymer electrolyte fuel cell in accordance with claim 1, 2 or 3, characterized by comprising:

supplying an oxidant gas and a fuel gas to said fuel electrode and said air electrode respectively; and outputting a current from said cell body with the polarity being inverted, thereby restoring the performance

of said cell.

[Claim 7] A method for restoring performance of the polymer electrolyte fuel cell in accordance with claim 1, 2 or 3, characterized by comprising

supplying oxygen to said air electrode, or supplying a pressurized gas to at least one of said air electrode and said fuel electrode in an amount not less than 1.5 times as much as that in the normal operation, thereby restoring the performance of said cell.

[Claim 8] A method for restoring performance of the polymer electrolyte fuel cell in accordance with claim 1, 2 or 3, characterized by comprising

injecting a cleaning solution into said air electrode and said fuel electrode through said gas flow paths of said separator plates, thereby restoring performance of said cell.

[Claim 9] The method for restoring performance of a polymer electrolyte fuel cell in accordance with claim 8,

characterized in that said cleaning solution is an acidic solution having a pH of less than 7.

[DETAILED DESCRIPTION OF THE INVENTION]

[0001]

[Technical Field to Which the Invention Belongs]

The present invention relates to fuel cells useful as commercial cogeneration systems and power generating devices for mobile units, specifically, polymer

electrolyte fuel cells using a polymer electrolyte and a method for restoring performance thereof.

[0002]

[Prior Art]

Fuel cells cause a fuel gas such as hydrogen and an oxidant gas such as air to electrochemically react at a gas diffusion electrode, thereby concurrently supplying electricity and heat. The fuel cells are classified into several types according to the kind of the electrolyte used therefor. In the case of the polymer electrolyte fuel cell employing a polymer as the electrolyte, an electrode paste prepared by mixing a dispersion of the above-described polymer electrolyte with a carbon powder carrying a platinum-based metal catalyst is applied onto both surfaces of a polymer electrolyte membrane comprising a skeleton of -CF<sub>2</sub>- as its main chain and a sulfonic acid attached to the terminal of its side chain, followed by drying to form an air electrode and a fuel electrode. On the outer surfaces of the air electrode and the fuel electrode, a porous conductive substrate such as a carbon paper, which is the electrode base material, is disposed as gas diffusion layers for air and a fuel gas. Herein, the electrode paste may be applied onto the above-described carbon paper, and the polymer electrolyte membrane may be bonded to this.

[0003]

Disposed on the outer surfaces of this are

conductive separator plates for mechanically fixing an assembly of the electrodes and electrolyte membrane and electrically connecting adjacent assemblies in series. Gas flow paths for supplying reactant gases to the electrodes and transporting water produced by the reaction of hydrogen and oxygen, residual gas and the like, are formed on the separator plates. A sealing member such as a gasket or a sealing agent is arranged on the peripheries of the gas flow paths, the electrodes and the like to prevent the reactant gases from directly mixing or from leaking outside.

[0004]

When this cell is used as a power generating device, it is common to laminate a plurality of the unit cells each comprising a polymer electrolyte layer, gas diffusion electrode layers, separator plates, gas flow paths and the like, in order to increase the output voltage. The respective gas flow paths supply a fuel gas such as hydrogen and air from outside through manifolds to the gas diffusion electrodes. Current generated at the reaction layers of the electrodes is collected at the electrode base material and is taken outside through the separator plates. As the separator plate, a carbon material having electrical conductivity, gas tightness and corrosion resistance is often employed. However, in terms of the moldability and cost-effectiveness as well as ease of thinning the separator, separators using a metal material such as

stainless steel are also being investigated.

[0005]

[Problem That the Invention Is to Solve]

Since the above-described polymer electrolyte has hydrogen ion conductivity when it contains water, the fuel gas to be supplied to the fuel cell is generally humidified. In addition, since the cell reaction produces water at the air electrode, water is always present within the cell. As a result, the ionic impurities, inorganic impurities and organic impurities contained in a carbon material, sealing material, resin material and metal material, each of which is the component of the cell, are eluted if the cell is operated for a long period of time. Moreover, air to be supplied from the outside of the cell contains air pollutants such as trace amounts of nitrogen oxides or sulfur oxides, and the fuel gas is occasionally contaminated with trace amounts of metal oxides contained in a hydrogen purifying device. Such impurities are accumulated in the polymer electrolyte membrane, catalytic reaction layers at the air electrode and fuel electrode and the like, leading to a reduction in the conductivity of the polymer electrolyte as well as the catalytic activity. Consequently, the cell performance is gradually degraded during a long operation of the cell. Additionally, in the case where a metal is used for the separator plate, metal ions eluted therefrom cause a further damage to the

electrolyte membrane, the catalytic reaction layers and the like.

[0006]

[Means for Solving the Problem]

In order to solve the foregoing problems, the polymer electrolyte fuel cell of the present invention is characterized by comprising (a cell body composed of laminated unit cells, each comprising a polymer electrolyte membrane, a fuel electrode and an air electrode sandwiching the polymer electrolyte membrane therebetween and a pair of separator plates having gas flow paths for supplying and discharging an oxidant gas to and from the air electrode and supplying and discharging a fuel gas to and from the fuel electrode);

means for supplying and discharging the oxidant gas and the fuel gas to and from the cell body; and

means for controlling output of a current generated in the cell body; and

means for restoring performance of the cell.

[0007]

Herein, the separator plate may contain a metal material.

[0008]

The above-described fuel cell is also characterized in that a contaminant ion accumulated in the cell body is discharged to the outside of the cell body with the means

for restoring performance of the cell.

[0009]

Further, the method for restoring the polymer electrolyte fuel cell of the present invention is characterized by comprising

operating the polymer electrolyte fuel cell in a loaded current mode different from that of a normal operation for a predetermined time, thereby restoring performance of the cell.

[0010]

Herein, it is effective that the loaded current mode different from that of the normal operation is either an operation mode at a current of not less than 1.5 times as high as that in the normal operation or an operation mode at a current giving an output voltage per unit cell of not more than 0.2 V.

[0011]

Further, the above-described method is characterized by comprising

supplying an oxidant gas and a fuel gas to the fuel electrode and the air electrode respectively; and

outputting a current from the cell body with the polarity being inverted, thereby restoring the performance of the cell.

[0012]

The above-described method is also characterized

by comprising

supplying oxygen to the air electrode, or supplying a pressurized gas to at least one of the air electrode and the fuel electrode in an amount not less than 1.5 times as much as that in the normal operation, thereby restoring the performance of the cell.

[0013]

Further, the above-described method is characterized by comprising

injecting a cleaning solution into the air electrode and the fuel electrode through the gas flow paths of the separator plates, thereby restoring performance of the cell.

[0014]

Herein, it is effective that the cleaning solution is an acidic solution having a pH of less than 7.

[0015]

[Mode for Embodying the Invention]

The ionic conductivity of the electrolyte used in the above-described polymer electrolyte fuel cell is achieved by hydrogen ions of the sulfonic group attached to the terminal of the polymer's side chain, which is pendant from the polymer's main chain. However, in the case where metal ions such as iron ions or sodium ions are present as impurities, these replace hydrogen ions to reduce the ionic conductivity of the electrolyte membrane. Further, since

the metal ions entered into the electrolyte have different hydrated states from those of hydrogen ions, the water content of the electrolyte is decreased, thereby reducing the ionic conductivity of the electrolyte membrane. Such reduction in the ionic conductivity and decrease in the water content not only increase the direct current resistance of the cell, but also decrease the reaction area of the catalytic layer in the electrode, thus causing a further degradation of the cell performance. Moreover, the above-described metal ions adhere to the surface of the catalyst, or seal the catalyst by forming oxides to degrade the cell performance. Furthermore, sulfur oxides, which are anionic impurities, poison the catalyst to degrade the cell performance, and nitrogen oxide ions and carboxylic acid ions, which are acidic substances, corrode and denaturalize the components.

[0016]

In the normal operation, these contaminant ions are present in high concentrations at a specific site in the cell. For example, the above-described metal ions are distributed in high concentration at the interface between the electrolyte membrane and the electrode layers or in the polymer electrolyte kneaded into the electrode. The acidic substances derived from the anionic impurities are gradually accumulated on the surfaces of the electrode base material serving as the gas diffusion layer and of the

separator plate. Since these metal ions, cationic impurities, and anionic impurities are not discharged to the outside of the cell in the normal operation, they become a cause of gradual degradation of the cell performance.

[0017]

The ionic impurities present in the cell are classified into those that are easy to move and those that are not, according to their ionic species. However, they both move along with a current passing through the cell, at a constant rate. Taking advantage of this fact, if the cell is operated at a current density of not less than 1.5 times as high as that in the normal operation (e.g. rated operation), the accumulated contaminant ions change the distributions thereof and are expelled from the electrolyte to be mixed with water produced by the electrode reaction, whereby it is possible to discharge them to the outside of the cell.

[0018]

In addition, it is possible to discharge the contaminant ions by switching the gases to be supplied to the fuel electrode and the air electrode and reversing the direction of the current and thereby transporting the ions opposite to the direction in which they entered.

[0019]

Further, it is possible to promote the transport

and discharge of the contaminant ions by pressurizing the reactant gases, or by using oxygen as the oxidant gas.

[0020]

Further, since the contaminant ions in the electrolyte are discharged to the outside by being replaced by hydrogen ions, it is possible to discharge them to the outside by washing the electrolyte, electrode and the like, with an acidic solution.

[0021]

[Working Examples]

In the following, preferred examples of the present invention will be concretely described.

[0022]

(Example 1)

An acetylene black carbon powder carrying 25 wt% of platinum particles having a mean particle size of approximately 30 Å was used as a catalyst for reaction electrode. A dispersion of this catalyst powder in isopropanol was mixed with a dispersion of a powder of perfluorocarbon sulfonic acid in ethyl alcohol to give an electrode paste.

[0023]

Meanwhile, a carbon paper having a thickness of 300 µm was immersed in an aqueous dispersion of polytetrafluoroethylene (PTFE), and this was subjected to drying to give a water-repellent porous electrode base

material. The above-described electrode paste was applied onto one surface of this porous electrode base material, followed by drying to give an electrode. Next, a polymer electrolyte membrane was sandwiched by a pair of the above electrodes, with the surface applied with the electrode paste facing inwards, and this was hot-pressed at a temperature of 110°C for 30 seconds, thereby fabricating an electrolyte membrane and electrode assembly (MEA). Herein, a membrane of perfluorocarbon sulfonic acid (Nafion manufactured by Du Pont), having a thickness of 50  $\mu\text{m}$ , was used as the polymer electrolyte membrane.

[0024]

In addition to the above-described carbon paper, a carbon cloth obtained by weaving carbon fiber, which is a flexible material, and a carbon felt obtained by molding a mixture of carbon fiber and carbon powder with an organic binder may also be used as the porous electrode base material.

[0025]

Next, as the separator plate, a resin-impregnated carbon plate was used, which had been obtained by cold press-molding a carbon powder material, impregnating this with a phenolic resin and then curing it to improve the gas sealing property, and a gas flow path was formed on this by cutting. Disposed on the periphery of the gas flow path were manifold apertures for supplying and discharging gases

and for supplying and discharging a cooling water to control the temperature of the cell. Further, in addition to the above-described carbon separator, a metal separator plate was prepared, which had been obtained by forming the gas flow paths and the manifold apertures on a metal plate made of SUS 304.

[0026]

A gasket made of silicone rubber serving as the gas sealing material was disposed on the periphery of the MEA having an electrode surface area of 25 cm<sup>2</sup> with current collecting plates made of SUS 304 sandwiching the MEA, and the whole was clamped from both ends, while being compressed at a pressure of 20 kgf/cm<sup>2</sup>.

[0027]

In practical use, a plurality of unit cells is generally laminated, with separator plates having a cooling water flow path formed thereon interposed therebetween. However, assuming that the above-described contaminant ions are hardly transported among different cells, the evaluation was made using a unit cell in the present examples. A gas supply system for supplying humidified reactant gases to the air electrode side and fuel electrode side, an electricity output system for setting and controlling the output of a loaded current to be taken out from the cell and a heat adjustment system for adjusting the cell temperature to utilize an exhaust heat were

attached to each of the unit cells fabricated as above, thereby obtaining polymer electrolyte fuel cells of the present examples.

[0028]

Each of the fuel cells fabricated in the above-described manner was operated with the following operational condition defined as the normal mode, and the cell whose performance had been degraded from the initial performance as a result of the operation, was used to evaluate the effectiveness of the method for restoring performance in accordance with the present invention.

First, the output current density was set at  $0.6 \text{ A/cm}^2$ . Next, the gas utilization rate, i.e., an index representing the ratio of the gas actually participating in the electrode reaction to the supplied fuel gas and oxidant gas, was set at 70% at the fuel electrode side and 30% at the air electrode side. Also, the cooling water was adjusted such that the cell temperature became  $75^\circ\text{C}$ . Then, pure hydrogen and air were used as the gases to be supplied, the pressure of the air supplied was set at  $0.2 \text{ kgf/cm}^2$ , that of hydrogen was set at  $0.05 \text{ kgf/cm}^2$ , and the outlet of the gas was opened to the atmosphere.

[0029]

As a result of operating the cell system under this condition, the performance of each of the cell employing carbon as the separator plate and the cell

employing SUS 304, started to be degraded after a continuous operation of 500 hours. At this time, the current was increased to  $0.8 \text{ A/cm}^2$ , and each cell was operated under this condition for 20 hours. Thereafter, each cell was operated with the current density being decreased to  $0.6 \text{ A/cm}^2$  again; however, no significant improvement was observed in the performance. Therefore, the current was increased to  $1.0 \text{ A/cm}^2$  once again, and each cell was operated under this condition for 20 hours to attempt to remove and discharge the contaminant ions and thereby restoring the cell performance. Further, a similar attempt was made at  $1.5 \text{ A/cm}^2$  and  $2.0 \text{ A/cm}^2$  to remove and discharge the contaminant ions. The results of these continuous cell tests are shown in FIG. 1.

[0030]

Referring to FIG. 1, in the case where the current density was increased to  $1.0 \text{ A/cm}^2$ , the cell voltage of the cell using the carbon separator was restored, from 570 mV to 590 mV, and that of the cell using the separator made of SUS 304 was restored, from 530 mV to 580 mV. Similarly, in the case where the current density was increased to  $1.5 \text{ A/cm}^2$  and  $2.0 \text{ A/cm}^2$ , the cell voltage of each cell was improved.

[0031]

By an analysis of the water discharged from each cell when the output current was increased by the above-

described method, iron ions were detected in the cell using the separator made of SUS 304, and a phenol component was detected in the cell using the carbon separator. This result demonstrated that the contaminant ions accumulated in the cell due to the long operation could be removed and discharged by the method of the present invention and thereby restoring the cell performance.

[0032]

In the foregoing, it was confirmed that the initial object could be achieved by changing the current density and thereby restoring the degraded performance of the cell caused by the continuous operation. Then, as a method for restoring the cell performance, the loaded current was increased to maintain the output voltage of each unit cell at not more than 0.2 V for a predetermined time and then the operation was returned to the normal mode. As a result, it was found that it was also possible to restore the output voltage as described above by using this method.

[0033]

Next, a method for restoring the cell performance was conducted by reversing the direction of the output current of the cells whose voltage had been dropped after a continuous operation of 500 hours. More specifically, air was supplied to the fuel electrode side, to which hydrogen was supplied in the normal operation (output current = 0.6

$\text{A}/\text{cm}^2$ ), and hydrogen was supplied to the air electrode side, to which air was supplied in the normal operation, and each cell was operated at  $0.6 \text{ A}/\text{cm}^2$  for 20 hours in this condition, with the direction of the output current being reversed. Thereafter, the operation was returned to the normal mode. It was found that, by conducting such method, the voltage of the cell using the carbon separator was restored, from 570 mV to 585 mV, and that of the cell using the separator made of SUS 304 was restored, from 530 mV to 565 mV.

[0034]

As described above, by conducting the method involving changing the magnitude and direction of the loaded current and the method involving reversing the supplied gases, it was possible to discharge the contaminants accumulated in the cell by mixing them into an exhaust gas and exhaust water, thereby restoring the cell performance.

[0035]

It was also possible to restore the cell performance by changing the direction of the supplied gases, that is, by supplying the reactant gases (air and hydrogen) from their respective discharge ports in the normal operation. Moreover, it was found that this effect of restoring the cell performance could be enhanced by supplying pure oxygen in place of air and using a

pressurized reactant gas.

[0036]

(Example 2)

Next, with the use of the same cells as those fabricated in Example 1, attempt was made to restore the cell performance by forcibly washing the cell with degraded performance to reduce the concentration of contaminant ions present in the cell.

[0037]

First, the above-described fuel cells were continuously operated for 500 hours in the normal operation mode as described in Example 1, and the operation was suspended when the cell voltage dropped from the initial voltage. Next, each of the cells was boiled in pure water for one hour to circulate the boiling pure water inside the cell through the gas flow paths for supplying the reactant gases. After this step, each cell was operated in the normal operation mode once again; consequently, the cell voltage of the cell using the carbon separator was restored, from 570 mV to 580 mV, and that of the cell using the separator made of SUS 304 was restored, from 530 mV to 555 mV.

[0038]

(Example 3)

While boiling water was used for washing the cells in the method described in Example 2, diluted

sulfuric acids having the respective pHs of 2 and 1 were used in the present examples. Each of the same fuel cells as those fabricated in Example 1 was operated in the same normal operation mode as in Example 1, and the operation was suspended; then, the respective diluted sulfuric acids were supplied, through a tube, to each of these cells from reactant gas supply ports (at the air electrode and fuel electrode sides), and discharged from the corresponding discharge ports. After washing with the diluted sulfuric acid for two hours, pure water was supplied to sufficiently wash each cell until the pH of the cleaning water discharged from the discharge port became 5 or more.

[0039]

After this step, each cell was operated in the normal operation mode once again; consequently, the cell voltage of the cell using the carbon separator was restored, from 580 mV to 588 mV, and that of the cell using the separator made of SUS 304 was restored, from 555 mV to 572 mV.

[0040]

In the above-described methods, weakly acidic diluted sulfuric acids were used for the cleaning solution; on the other hand, in the case of using a weakly alkaline cleaning solution, i.e., a cleaning solution having a pH of approximately 9, no significant restoration was observed, although it was confirmed that a little effect was achieved

by the washing. The results of the foregoing are shown in FIG. 2.

[0041]

As such, it was found that it was possible to restore the cell performance by washing the inside of the cell with the cleaning solution. It was also confirmed that, in such a case, the higher the temperature at which the washing was conducted, the higher the effect of restoring became. It was also proved that it was possible to enhance the restoration of the cell voltage by concurrently using the restoring method of Example 1 involving the high current density operation and the washing of the cell with the weakly acidic cleaning solution. Further, it was confirmed that a similar effect could be achieved by using a diluted acetic acid or ammonium sulfate for the weakly acidic cleaning solution.

[0042]

The effect of the present invention demonstrated in Examples 1, 2 and 3 in consideration of the difference in the materials forming the separator that was the component of the cell body, is summarized as follows. Although the performance of the cell using the metal separator was degraded owing to the metal ions eluted from the separator during the long operation, it was possible to restore the cell performance by removing the metal ions accumulated in the cell by the operation at a high current

and the washing with the weakly acidic cleaning solution.

[0043]

On the other hand, in the case of the cell using the carbon separator as a component, the metal ions or various cations were not eluted as much as those in the case of the cell using the metal separator; however, trace amounts of iron and calcium were found to be contained in the separator through an ashing analysis. Accordingly, although the performance of the cell was not degraded as significantly as that of the cell using the metal separator, it was degraded to some extent during the long operation, owing to the contained metal ions. In addition, it was considered that the performance decreased by approximately 30 mV after the continuous test of 500 hours because of organic materials eluted from the resin, which had been added for improving the gas tightness of the carbon separator, as well as trace amounts of sulfur compounds and nitrogen oxides contained in air. The restoring method of the present invention was effective even for the cell using the carbon separator.

[0044]

To summarize the enabling requirements of the present invention, they include: a polymer electrolyte fuel cell having, in its electricity output system, means for adjusting a loaded current and an output voltage, or having in its gas supply system, a means capable of supplying a

cleaning solution to a gas flow path to directly wash the inside of the cell; a cell operating method involving controlling a loaded current and an output voltage, or washing the inside of the cell, after the elapse of a predetermined period, or after a predetermined period of operation, or after the cell performance has been degraded; and a device such as a system having a fuel reforming system, controlling system, charger system and the like in addition to the above-mentioned cell, gas supply system, heat adjustment system and electricity output system; for example, an electric vehicle equipped with a fuel cell, a cogeneration system and a portable power source system.

[0045]

**[Effects of the Invention]**

According to the present invention, it is possible to effectively restore a degraded performance of a polymer electrolyte fuel cell caused by a long operation, thereby realizing a high durability.

**[BRIEF EXPLANATION OF THE DRAWINGS]**

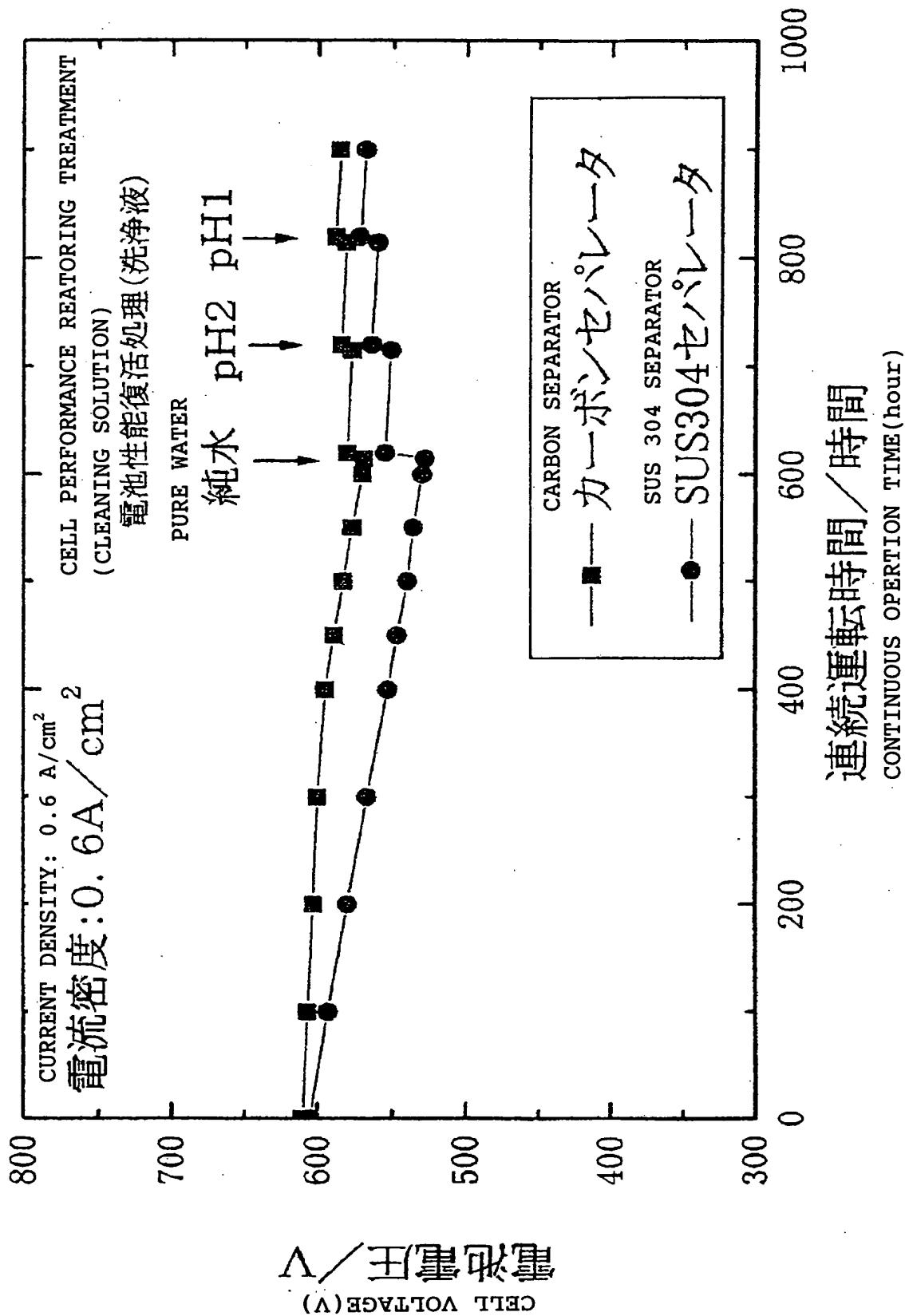
**[FIG.1]**

A graph showing the performance restoration of the cells in Example 1 of the present invention.

**[FIG. 2]**

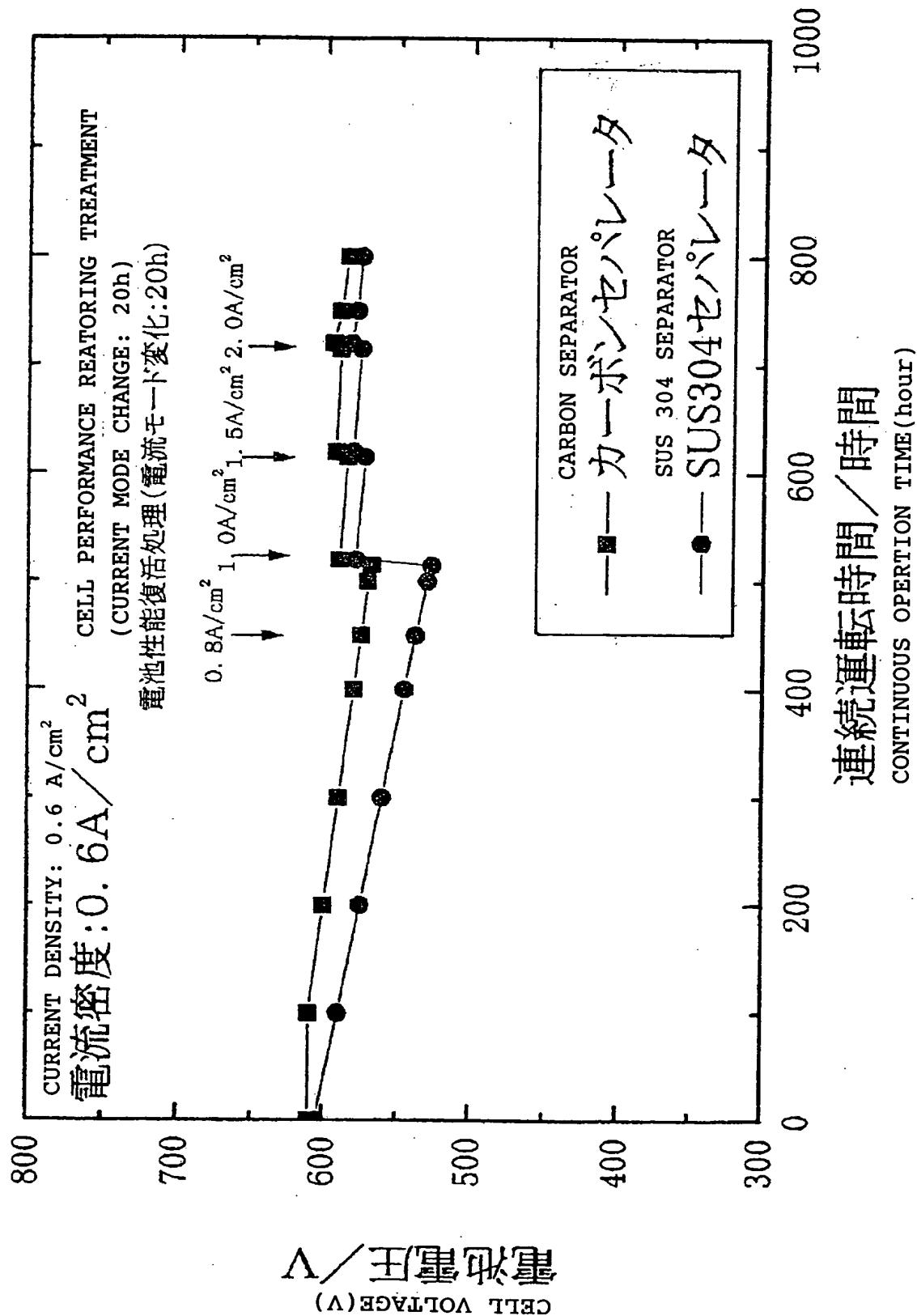
A graph showing the performance restoration of the cells in another example of the present invention.

【図2】 [FIG. 2]



【書類名】 図面 [DOCUMENT NAME] Drawings

【図 1】 [FIG. 1]



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(Translation)

[DOCUMENT NAME] Abstract

[ABSTRACT]

[OBJECTIVE] In a polymer electrolyte fuel cell, metal ions or contaminants, which have been eluted from a component such as a separator, accumulate inside the cell during a long operation, resulting in degradation of the performance and durability.

[SOLVING MEANS] To apply a high current density load to a cell with degraded performance, or to reverse the direction of the current, or to supply an acidic water having a pH of not more than 7 to the gas flow path.

[SELECTED DRAWING] FIG. 2